Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड़ नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 10177 (1982): Ice cream cups and lids [CHD 15: Paper and its products]

"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"
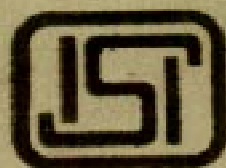
BLANK PAGE

Indian Standard

SPECIFICATION FOR
ICE CREAM CUPS AND LID

UDC 621·798·147 [676·828] : 663·674

© *Copyright* 1982

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR
ICE CREAM CUPS AND LIDS

Paper and Flexible Packaging Sectional Committee, MCPD 14

Chairman	*Representing*
SHRI K. VISWANATHAN	Metal Box India Ltd, Calcutta

Members

SHRI K. R. NARASIMHAN (*Alternate* to Shri K. Viswanathan)	
DR S. R. AGARWAL	Glaxo Laboratories (India) Ltd, Bombay
SHRI S. M. KHAN (*Alternate*)	
SHRI A. B. AJMERA	Card Board Box Manufacturing Co, Calcutta
SHRI C. B. AJMERA (*Alternate*)	
DR R. P. ANEJA	National Dairy Development Board, Anand
SHRI G. K. BHAIA	Indian Plastics Federation, Calcutta
SHRI VIDYA CHARAN BHARGAVA	Ministry of Agriculture, Directorate of Plant Protection, Quarantine & Storage, Faridabad
SHRI S. K. GHOSH (*Alternate*)	
SHRI L. N. BURMAN	Indian Paper Makers Association, Calcutta
SHRI P. L. BURMAN	Indian Paper Mills Association, Calcutta
SHRI D. S. CHADHA	Directorate General of Health Services (Central Committee for Food Standards), New Delhi
SMT DEBI MUKHERJEE (*Alternate*)	
SHRI S. CHAKRABARTI	Hindustan Lever Ltd, Bombay
SHRI V. DHAWAN (*Alternate*)	
SHRI B. D. CHATTERJEE	Indian Petrochemicals Corporation Ltd, Vadodara
SHRI P. R. SESHAN (*Alternate*)	
SHRI T. GANGADHARAN	Indian Explosives Ltd, Calcutta
SHRI R. KRISHNAN (*Alternate*)	
SHRI P. S. HARIHARAKRISHNAN	The Travancore Rayons Ltd, Rayonpuram
SHRI BHAGVAN BALAJI SINGH (*Alternate*)	
DR R. JAYARAM	Federation of Biscuit Manufacturers of India, Delhi
JOINT DIRECTOR (T R A F F I C COMMERCIAL RATES)	Railway Board, Ministry of Railways, New Delhi
SHRI S. K. KESHAVA	I. T. C. Ltd, Calcutta
SHRI Y. C. DEVESHWAR (*Alternate*)	
SHRI A. J. KOTHARI	Larsen & Toubro Ltd, Bombay
SHRI R. P. SOOCHAK	

(Continued from page 1)

Members	Representing
SHRI J. S. MATHARU	Directorate General of Technical Development, New Delhi
SHRI V. B. SAXENA (*Alternate*)	
SHRI A. L. MUTHIAH	India Packaging Products Pvt Ltd, Bangalore
SHRI A. L. ANNAMALAI (*Alternate*)	
SHRI M. S. NAIK	Pesticides Association of India, New Delhi
DR A. N. NAYER	Packaging and Wood Products Consultancy Service, New Delhi
SHRI SANJAY K. NAYER (*Alternate*)	
SHRI P. V. NARAYANAN	Indian Institute of Packaging, Bombay
SHRI A. K. SENGUPTA (*Alternate*)	
DR S. Y. PANDIT	Bayer (India) Ltd, Bombay
DR P. V. RAIKAR (*Alternate*)	
SHRI M. K. RAMASAMI	Jayant Packaging Pvt Ltd, Madras
SHRI R. MUTHIAH (*Alternate*)	
SHRI M. K. RAMASAMI	Federation of Corrugated Box Manufacturers of India, Bombay
SHRI S. S. KEJRIWAL (*Alternate*)	
SHRI C. V. N. RAO	Office of the Development Commissioner, New Delhi
SHRI G. V. RAO	Directorate of Standardization, Ministry of Defence (R & D), New Delhi
SHRI O. P. SRIVASTAVA (*Alternate*)	
SHRI J. C. SHAH	Federation of Associations of Small Industries of India, New Delhi
SHRI D. CHADHA (*Alternate*)	
SHRI PRAFULL J. SHETH	Solid Containers Ltd, Bombay
SHRI V. S. JOSHI (*Alternate*)	
SHRI M. K. LAHERI (*Alternate*)	
SHRI S. SUNDER	India Foils Ltd, Calcutta
SHRI S. P. CHATTERJEE (*Alternate*)	
SHRI R. C. TANDAN	Forest Research Institute & Colleges, Dehra Dun
SHRI D. V. VARMA	Ministry of Defence (DGI)
SHRI NIRMAL SINGH (*Alternate*)	
SHRI P. S. DAS, Director (MCPD)	Director General, ISI (*Ex-officio Member*)

Secretary

SHRI A. R. GULATI
Deputy Director (MCPD), ISI

Cartons Subcommittee, MCPD 14 : 1

Convener	
SHRI T. V. S. MANIAN	India Coated Cartons Pvt Ltd, Bangalore
Members	
SHRI R. M. MUTHIAH (*Alternate* to Shri T. V. S. Manian)	
DR S. R. AGARWAL	Glaxo Laboratories (India) Ltd, Bombay
SHRI S. K. KHAN (*Alternate*)	

2

Indian Standard

SPECIFICATION FOR
ICE CREAM CUPS AND LIDS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 April 1982, after the draft finalized by the Paper and Flexible Packaging Sectional Committee had been approved by the Marine, Cargo Movement and Packaging Division Council.

0.2 Paper cups are largely used for ice cream packing. The basic factors taken into consideration while laying down the requirements of these cups were the quality of the paperboard, Coating wax, printing inks, adhesives etc to be acceptable as food grade and should not impart any off-flavour or taint to the contents during storage.

0.3 Standard of Weight and Measure (Packaged Commodities) Rules, 1977 specify the quantity of the ice cream to be packed in cups as 25 g, and thereafter in multiples of 25 g. If the net quantity is declared by volume, the ice cream content may be indicated by the same number in ml or litres, as the case may be. Normally paper cups of 50, 75 and 100 ml are used for packing of ice creams. The product being such that it is not feasible to specify the shape or dimensions of the cups, Fig. 1 and 2 of 50 and 100 ml cups respectively given in the standard are therefore, for illustrative purpose only and should not be taken as the recommended design.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

---

1. SCOPE

1.1 This standard specifies the requirements for waxed paper cups with lids for packing of ice cream.

---

*Rules for rounding off numerical values (*revised*).

2. CAPACITY

2.1 Quantity of ice cream packed in paper cups shall be 50 g, 75 g or 100 g if packed by mass. If net quantity is declared by volume the same number in millilitres shall be packed. The nominal capacity of the cup shall be designated by the quantity of ice cream to be packed into it, for example 50 ml, 75 ml or 100 ml.

3. REQUIREMENTS

3.1 Dimensions — The dimensions of cups shall be as specified by the purchaser. Figures 1 and 2 illustrate the shape and dimensions of 50 ml and 100 ml cups respectively.

3.1.1 A tolerance of ± 1 mm shall apply to the specified diameter and height of the cup.

3.2 Material

3.2.1 *Paper* — The paper used for the manufacture of cups and lids shall be a good quality material made from virgin chemical wood pulp and shall conform to IS : 1776-1961*. It shall be capable of being waxed.

3.2.1.1 *Substance of paper* — The substance of paper used for cups and lids shall be of 140 g/m² min. in unwaxed condition.

3.2.1.2 *Stiffness* — The stiffness of the waxed board shall be not less than 110 g cm in machine direction and 40 g cm in cross direction, when determined by the method given in 7 in IS : 1060 (Part III)-1969†.

3.2.2 *Ink* — The ink and other additives used for printing of the cups and lids shall be commercially free from toxic metals, such as, lead and chromium and free from residual odour. The residual odour shall be tested after waxing, in accordance with the method prescribed in 7 of IS : 4006 (Part II)-1972‡. The ink shall be suitable for food packaging and shall be fast to light and wax. In order to prevent ink set-off, a coating of an edible starch may be applied to the surface of the printed board by spraying.
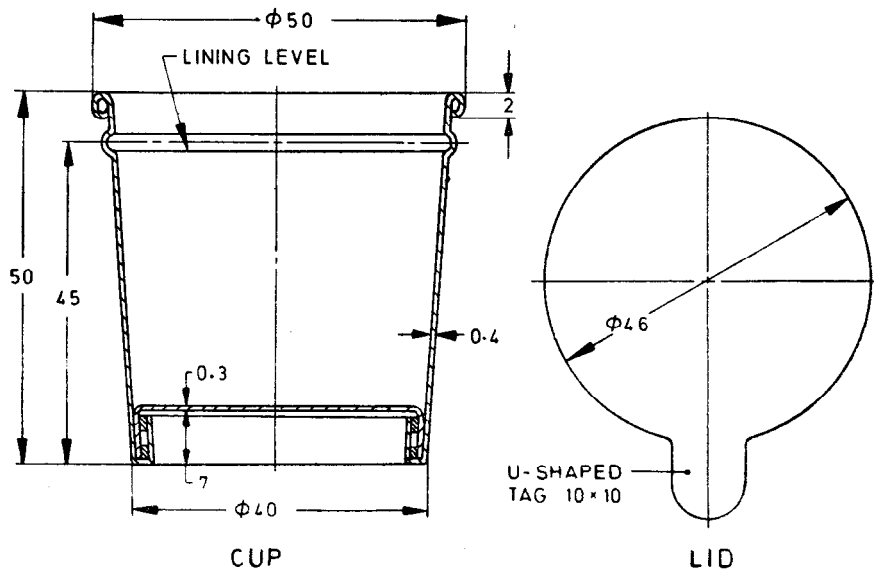
3.2.3 *Wax* — The wax for coating the board shall be paraffin wax conforming to Type 1 of IS : 4654-1974§ with melting point not less than 60°C.

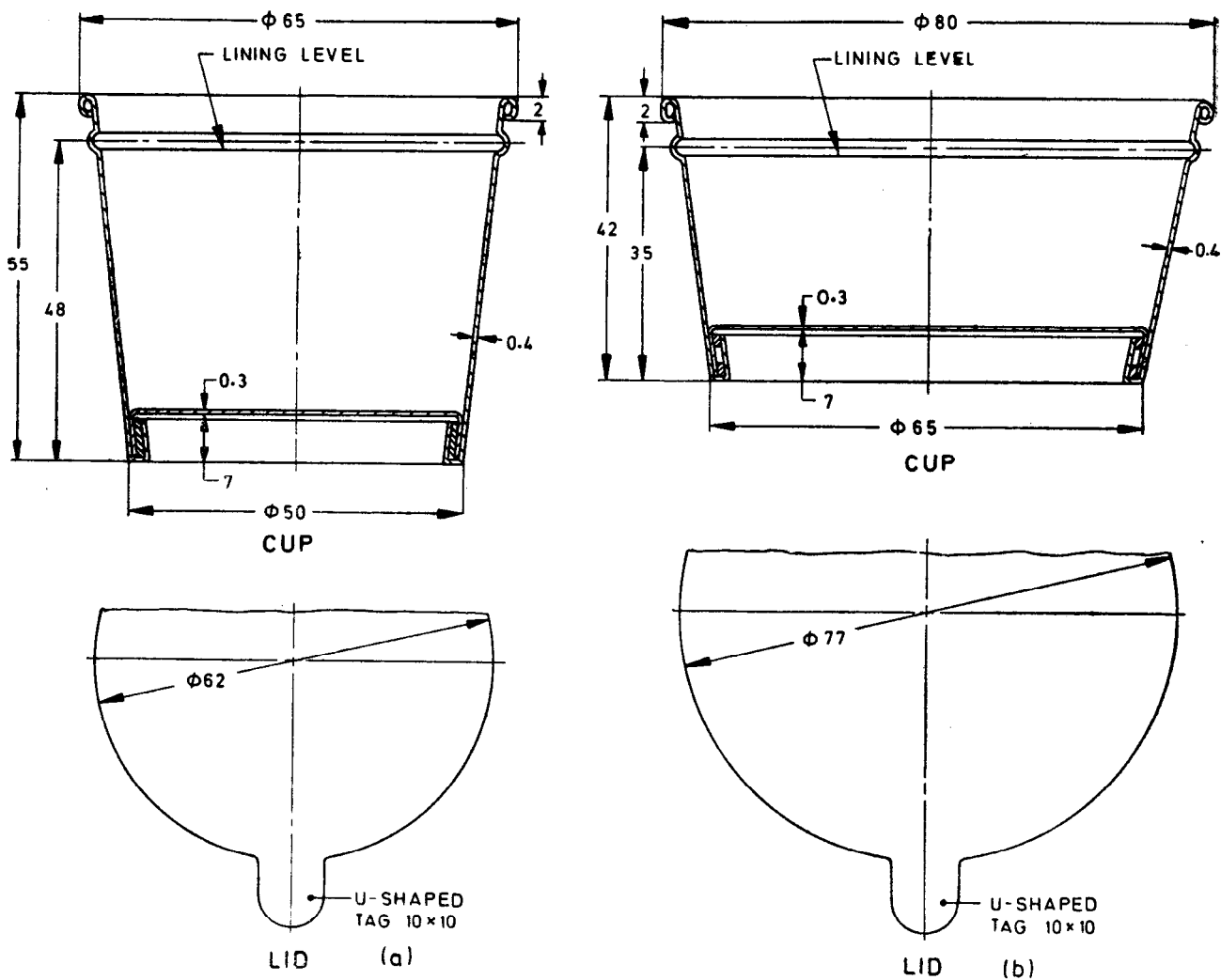*Specification for folding box board, uncoated.
†Methods of sampling and test for paper and allied products (Part III).
‡Methods of test for paper and pulp based packaging materials (Part II).
§Specification for paraffin wax (*first revision*).

4

All dimensions in millimetres.

FIG. 1   50 ml ICE CREAM CUP

All dimensions in millimetres.

FIG. 2   100 ml ICE CREAM CUPS

As in the Original Standard, this Page is Intentionally Left Blank

3.2.4 *Adhesive* — The adhesive used in the manufacture of cups shall be resistant to deep-freeze condition of — 35°C and shall be non-toxic.

3.3 Performance

3.3.1 *Wax Content* — The cup and lid shall be evenly coated with wax on both sides as far as possible. The total wax content of the board shall be 30·0 g/m² minimum when determined by the method given in Appendix A.

3.3.2 *Odour and Taint* — The composition of the board and paper, ink, coating material and adhesives shall not give rise to any deleterious effect on the flavour odour or appearance of the product packed [*see* 7 of IS : 4006 (Part II)-1972*].

3.3.3 *Toxicity* — The composition of the board, ink, coating material shall be such as not to introduce any substance into the product packed, injurious to health or which might otherwise lead to infringement of the Preventing of Food Adulteration Act, 1954 and the Regulations made under this Act.

3.3.4 *Resistance to Deep-Freezing* — The printed waxed cups shall withstand deep-freeze conditions of — 35°C for a period of 30 days.

3.3.5 *Leakproofness* — The waxed cups shall be leakproof. There shall be no cracks on the outer curling of the cups and the base curling shall be uniform. Glue spots shall be absent where the cups are seamed.

3.3.5.1 To test the leakproofness of cups the sample cups shall be filled with water and checked after 5 minutes for any leakage.

4. PACKING AND MARKING

4.1 Packing — The cups shall be packed in either polyethlyene bags or in suitable protective wrapper and then packed in protective containers as agreed to between the supplier and the purchaser. The cups and lids shall be stored and transported under hygienic conditions.

4.1.1 The empty cups may also be marked with the ISI Certification Mark.

NOTE 1 — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Mark) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity

---

*Methods of test for paper and pulp based packaging materials (Part II).

to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

NOTE 2 — The empty cups and lids if covered under the ISI mark scheme, shall be marked in such a way that the marking for the cups is not in anyway construed for the marking on the contents packed into them.

4.1.2 *Transit Containers* — Each protective transit container shall be suitably marked to indicate the following information in respect of the cartons packed in it:

- a) Name or trade-mark of the manufacturer;
- b) Name of the product packed;
- c) Batch number; and
- d) Date of manufacture.

APPENDIX A

(*Clause* 3.3.1)

METHOD FOR DETERMINATION OF WAX CONTENT

A-0. PRINCIPLE

A-0.1 A known amount of waxed-board cuttings is extracted with carbon tetrachloride in a Soxhlet apparatus. The extract is evaporated to dryness and the residue re-extracted with alcoholic potassium hydroxide and evaporated to dryness. The dried mass is then dissolved in water and petroleum ether and the two layers are separated. The petroleum ether layer is evaporated to dryness and the residue weighed.

A-1. APPARATUS

A-1.1 Soxhlet Apparatus

A-1.2 Separating Funnel

A-2. REAGENTS

A-2.1 Carbon Tetrachloride

A-2.2 Alcoholic Potassium Hydroxide Solution — Approximately 0·5 N.

A-2.3 Petroleum Ether — Boiling range 40 to 60°C.

A-3. PROCEDURE

A-3.1 Weigh accurately about 10 g of the specimen, cut it into strips and give numerous small crosswise folds to each strip. Extract the strips in a Soxhlet apparatus with carbon tetrachloride for 4 hours or until the wax is all removed.

A-3.2 Evaporate the extract to dryness on a water-bath, add 25 ml of alcholic potassium hydroxide solution and again evaporate to dryness. Cool the residue, take it up with petroleum ether and water (*see Note*), and transfer to a separating funnel. Shake the contents of the funnel thoroughly and then allow the two layers to separate completely. Draw off the water layer into another separating funnel.

> NOTE -- The volume of petroleum ether should be about 25 ml and that of water about 150 ml. The water should contain a small amount of sodium chloride to prevent emulsification.

A-3.3 Re-extract the water layer one or more times, as may be found necessary, with fresh 25 ml aliquot of petroleum ether.

A-3.4 Wash the petroleum ether extract obtained as in **A-3.2** and **A-3.3** with fresh 100 ml aliquots of water until the water layer is perfectly clear. Addition of a concentrated solution of sodium chloride may be found necessary while washing.

A-3.5 Transfer the petroleum ether extract to a weighed evaporating dish or flask and evaporate to dryness. Dry at 103 $\pm$ 2°C for 1 hour and weigh.

A-4. CALCULATION

A-4.1 Calculate the wax content of the board by the following formula:

$$\text{Wax, percent by mass} = \frac{100\ (W_3 - W_2)}{W_1}$$

where

W_3 = mass in g of the evaporating dish or flask with residue (**A-3.5**),

W_2 = tare in g of the evaporating dish or flask (**A-3.5**), and

W_1 = mass in g of the specimen taken for the test (**A-3.1**).

AMENDMENT NO. 1 APRIL 1998
TO
IS 10177 : 1982 SPECIFICATION FOR
ICE CREAM CUPS AND LIDS

(*Page* 3, *clause* **0.3**) — Insert the following clause after **0.3** and renumber the subsequent clause:

'**0.4** A scheme for labelling environment friendly products to be known as ECO Mark has been introduced at the instance of the Ministry of Environment and Forests (MEF). The ECO Mark shall be administered by the Bureau of Indian Standards (BIS) under the *BIS Act,* 1986 as per the Resolution No. 71 dated 20 February 1991, published in the Gazette of the Government of India. For a product to be eligible for ECO Mark it shall also carry Standard Mark of BIS for quality, in addition to the compliance with the optional environment friendly (EF) requirements. For this purpose, the Standard Mark of BIS would be a single mark being a combination of the ISI Mark and the ECO logo. Requirements to be satisfied for a product to qualify for the BIS Standard Mark for ECO friendliness, will be included in the relevant published Indian Standard through an amendment. These requirements will be optional; manufacturing units will be free to opt for the ISI Mark alone also.

This amendment is based on the *Gazette Notification No.* 364 dated 7 September 1995 for packaging material/package (Part I Paper Board and Plastics Excluding Laminates) as environment friendly products, published in the Gazette of India.This amendment is, therefore, being issued to this standard to include EF requirements for ice cream cups and lids.'

(*Page* 7, *clause* **3.3.5.1**) — Insert the following new clauses after 3.5.5.1:

'3.4 Additional Requirements for ECO Mark

3.4.1 *General Requirements*

3.4.1.1 The product shall conform to the requirements for quality and performance prescribed under **3.1** to **3.3**.

3.4.1.2 The manufacturer shall produce to BIS, the environmental consent clearance from the concerned State Pollution Control Board as per the provisions of *Water*

Amend No. 1 to IS 10177 : 1982

(Prevention and Control of Pollution) Act, 1974 and *Air (Prevention and Control of Pollution) Act*, 1981 along with the authorization, if required under the *Environment (Protection) Act*, 1986 and the Rules made thereunder, while applying for ECO Mark. Additionally the manufacturers shall also comply with the provisions under *Prevention of Food Adulteration Act*, 1954 and the Rules made thereunder wherever necessary.

3.4.2 *Specific Requirements*

3.4.2.1 The material shall be of the following two types depending on the raw material used in the manufacture:

   a) *Type A* — Manufactured from pulp containing not less than 60 percent by mass of pulp made from materials other than bamboo, hardwood, softwood and reed.
   b) *Type B* — Manufactured from pulp made from 100 percent waste paper or agricultural/industrial waste.

3.4.2.2 The material if used for the packaging of food materials, shall be manufactured from virgin pulp and shall be free from dioxins. Printed surfaces of the paper shall not come into contact with the food and the maximum amounts of contaminants in paper intended to come into contact with food shall not exceed the limits prescribed in Table 1 when tested according to the methods given in Annex B.

Table 1 Limits of Contaminants in Paper
(*Clause* 3.4.2.2)

Contaminant	Paper Intended to Come into Contact with Dry Food	Paper Intended to Come into Contact with Wet Food and Food with Fatty Surface	Paper for Filtration
	(mg/kg of paper)	(mg/kg of paper)	(mg/kg of paper)
Cadmium (Cd)	—	0.5	0.5
Chromium (Cr^{6+})	—	0.1	0.1
Lead (Pb)	—	3.0	3.0
Mercury (Hg)	—	0.3	0.3
Pentachlorophenol (PCP)	0.05	0.05	0.05
Polychlorinated biphenyls (PCBs)	2.0	2.0	0.5

2

(Page 8, *clause* **4.1.2)** — Insert the following new clauses after **4.1.2**:

'**4.1.3** *Additional Requirements for ECO Mark*

4.1.3.1 For ECO Mark, ice cream cups and lids shall be packed in such packages which shall be recyclable/reusable or biodegradable.

4.1.3.2 The ice cream cups and lids may display in brief the criteria based on which the product has been labelled as environment friendly.

4.1.3.3 The ice cream cups and lids may be sold along with instruction for proper use and mode of safe disposal so as to maximise its performance and minimise wastage.

4.1.3.4 It shall be suitably marked on ice cream cups and lids that ECO Mark label is applicable only to the packaging material/package if content is not separately covered under the ECO Mark scheme.

NOTE — It may be stated that the ECO Mark is applicable to the product or packaging material or both.'

(Page 8, *Annex* A) — Insert the following text after Annex A:

ANNEX B

(Clause 3.4.2.2)

DETERMINATION OF CHROMIUM, LEAD, MERCURY, CADMIUM, PENTACHLOROPHENOL AND POLYCHLORINATED BIPHENYLS

B-1 DETERMINATION OF CHROMIUM (as Cr^{6+})

B-1.1 Principle

The hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution at a wavelength of 550 nm.

B-1.2 Apparatus

B-1.2.1 *Spectrophotometer*

Any spectrophotometer suitable for measurement at a wavelength of about 550 nm or photoelectric absorptiometer fitted with filters giving maximum transmission near 550 nm.

B-1.2.2 *Shaker*

Any shaker suitable for rotating/moving at 30 ± 2 rev/min.

B-1.3 Reagents

B-1.3.1 *Extraction Fluid* — Mix 5.7 ml of acetic acid in distilled water.

B-1.3.2 *Nitric Acid* — Concentrated.

B-1.3.3 *Sulphuric Acid* — Approximately 0.2 N.

B-1.3.4 *Diphenylcarbazide Solution*

Dissolve 0.25 g of diphenylcarbazide in 50 ml acetone. Store in a brown bottle. Discard when solution becomes discoloured.

B-1.3.5 *Stock Chromium Solution*

Dissolve 0.141 g $K_2Cr_2O_7$ in distilled water and dilute to 100 ml. One millilitre of this solution contains 500 μg of chromium (as Cr^{6+}).

B-1.3.6 *Intermediate Chromium Solution*

Take 10 ml of stock chromium solution and dilute to 1 000 ml with distilled water. One millilitre of this solution contains 5.00 μg of chromium (as Cr^{6+}).

B-1.3.7 *Standard Chromium Solution*

Take 10 ml of intermediate chromium solution and dilute to 1 000 ml with distilled water. One millilitre of this solution contains 0.05 μg of chromium (as Cr^{6+}).

B-1.3.8 *Indicator Paper* — Covering the *p*H range 0.5 to 1.5.

B-1.4 Procedure

B-1.4.1 *Preparation of Calibration Curve*

Into each of a series of ten 250-ml volumetric flasks, place the quantities of standard chromium solution as indicated below:

Standard Chromium Solution	Corresponding to Cr^{6+}
ml	µg
1.0	0.05
2.0	0.10
3.0	0.15
4.0	0.20
5.0	0.25
6.0	0.30
7.0	0.35
8.0	0.40
9.0	0.45
10.0	0.50

B-1.4.1.1 Add sulphuric acid to adjust the solution pH to 1.0 ± 0.3 in each flask and dilute to 100 ml. Add 2.0 ml diphenylcarbazide solution, mix thoroughly and wait for 10 minutes.

B-1.4.1.2 Carry out the measurement on the spectrophotometer or on a photoelectric colorimeter using appropriate filter with a 1-cm cell at a wavelength of 550 nm. As references use extraction fluid. Correct the absorbance readings of standard solution by subtracting absorbance of a reagent blank carried through the above method.

B-1.4.1.3 Construct a calibration curve by plotting corrected absorbance values against chromium content in microgram per 102 ml.

B-1.4.2 *Determination*

B-1.4.2.1 *Sample preparation*

Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

B-1.4.2.2 *Preparation of test solution*

Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for 18 ± 2 h using shaker rotating/moving at 30 ± 2 rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size. Add sufficient amount of aluminium sulphate and filter if any precipitate appears. Transfer the solution quantitatively to a volumetric flask of suitable capacity, dilute to the mark and mix.

B-1.4.2.3 According to the expected chromium content, take an aliquot portion of the test solution containing 10 to 100 µg of chromium to a 100-ml volumetric flask. Adjust the *p*H of the solution to 1.0 ± 0.3 by adding 0.2 N sulphuric acid. Dilute to 100 ml. Add 2 ml diphenylcarbazide solution, mix thoroughly, and wait for 10 minutes.

B-1.4.2.4 *Photometric measurement*

Carry out the photometric measurements of the test solution according to the methods given in **B-1.4.1.1**.

B-1.4.3 *Calculation*

By means of the calibration curve (*see* **B-1.4.1.3**) determine the quantity of chromium present:

$$\text{Chromium (as Cr}^{6+}\text{), ppm} = \frac{m \times D}{M}$$

where
- m = mass of chromium determined in the aliquot of the sample solution, µg;
- D = ratio of the volume of test solution to the volume of aliquot portion taken for the colour development; and
- M = mass of paper sample taken for testing, g.

B-2 DETERMINATION OF MERCURY (as Hg)

B-2.1 Principle

The flameless atomic absorption procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapour. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapour passes through a cell positioned in the light path of mercury hallow cathode lamp of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration and record.

B-2.2 Apparatus

B-2.2.1 *Atomic Absorption Spectrometer (AAS) and Associated Equipment*

Instrument settings recommended by the manufacturer shall be followed. Instruments designed specifically for the measurement of mercury using the cold vapour technique may be substituted for the AAS.

B-2.2.2 *Mercury Vapour Generation Assembly*

Consists of an absorption cell, peristaltic pump, flow meter, aeration tubing and a drying tube containing magnesium perchlorate.

B-2.2.3 *Mercury Hollow Cathode Lamp*

B-2.2.4 *Recorder/Printer/Display Meter*

Any multi-range variable recorder that is compatible with the UV detection system is suitable.

B-2.3 Reagents

B-2.3.1 *Sulphuric Acid* — Concentrated.

B-2.3.2 *Nitric Acid* — Concentrated.

B-2.3.3 *Stannous Chloride Solution*

Dissolve 25 g of stannous chloride ($SnCl_2$) in water containing 50 ml of concentrated hydrochloric acid and dilute to 250 ml. If a suspension forms, stir reagent continuously during use.

B-2.3.4 *Sodium Chloride — Hydroxylamine Sulphate Solution*

Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulphate ($NH_2OH)_2$ H_2SO_4 in distilled water and dilute to 100 ml.

B-2.3.5 *Potassium Permanganate Solution*

Dissolve 5 g of potassium permanganate in distilled water and dilute to 100 ml.

B-2.3.6 *Potassium Persulphate Solution*

Dissolve 5 g of potassium persulphate in distilled water and dilute to 100 ml.

B-2.3.7 *Stock Mercury Solution*

Dissolve 1.354 g of mercuric chloride in about 700 ml of distilled water. Add 10 ml of concentrated nitric acid and make up to 1 000 ml. One millilitre of the solution contains 1 mg of mercury as Hg.

7

B-2.3.8 *Standard Mercury Solution*

Prepare a series of standard mercury solutions containing 0 to 5 µg/l by appropriate dilution of stock mercury solution (**B-2.3.7**) with water containing 10 ml of concentrated nitric acid per litre. Prepare standards daily.

NOTE — Use mercury free distilled water for the preparation of reagents and standards.

B-2.4 Procedure

B-2.4.1 *Instrument Operation*

Follow the procedure of the manufacturer's operating manual. Connect the mercury vapour generating assembly as shown in Fig. 1.
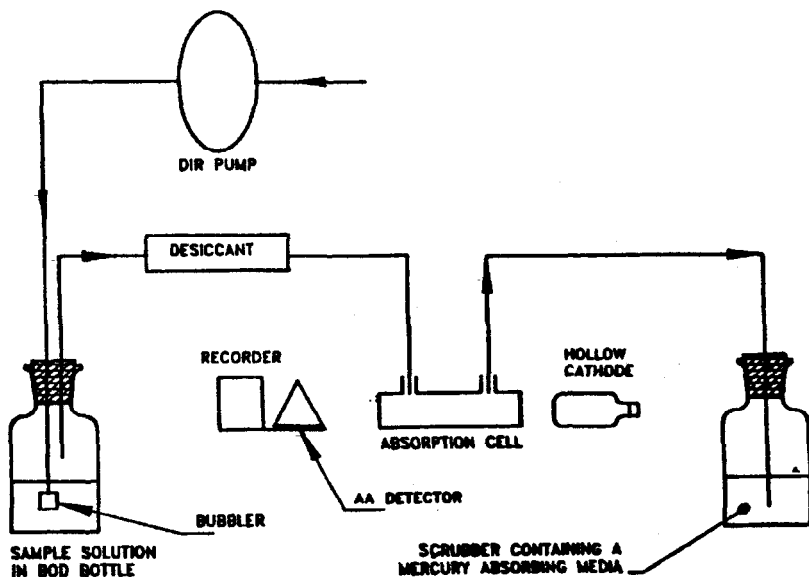

FIG. 1 SCHEMATIC ARRANGEMENT OF EQUIPMENT FOR MEASUREMENT OF
MERCURY BY COLD VAPOUR ATOMIC ABSORPTION TECHNIQUE

8

B-2.4.2 *Standardization*

Transfer 100 ml of each of the 1.0, 2.0 and 5.0 µg/l standard mercury solution and a blank of 100 ml water to 300 ml BOD bottles. Add 5 ml of concentrated sulphuric acid and 2.5 ml of concentrated nitric acid to each bottle. Add 15 ml of potassium permanganate solution to each bottle and let stand for at least 15 minutes. Add 8 ml of potassium persulphate ($K_2S_2O_8$) solution to each bottle and heat for 2 hours in a water bath at 95°C. Cool and add 6 ml of sodium chloride-hydroxylamine sulphate solution to reduce the excess permanganate. After decolourization add 5 ml of stannous chloride solution and attach the bottle immediately to the aeration apparatus forming a closed system. As mercury is volatilised and carried into the absorption cell, absorbance will increase to a maximum within a few seconds. As soon as recorder returns approximately to the base line, remove stopper holding the aeration frit from the reaction bottle and replace with a bottle containing distilled water. Flush the system for a few seconds and run the next standard in the same manner. Construct a standard calibration curve by plotting absorbance (peak height) versus mercury concentration in µg.

B-2.4.3 *Determination*

B-2.4.3.1 *Sample preparation*

Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

B-2.4.3.2 *Preparation of test solution*

Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for 18 ± 2 h using shaker rotating/moving at 30 ± 2 rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size. Transfer the solution quantitatively to a volumetric flask of suitable capacity, dilute to the mark and mix.

B-2.4.3.3 According to the expected mercury content, take an aliquot portion of the test solution containing not more than 5 µg/l of mercury to a 300 ml BOD bottle and treat as in **B-2.4.2**.

B-2.5 **Calculation**

Determine peak height of sample from recorder chart, read mercury value from standard curve and determine the mercury content of the sample using the following formula:

$$\text{Mercury (as Hg), ppm} = \frac{C \times V}{M \times 1\,000}$$

where

C = concentration of mercury from the calibration curve;

V = volume of test solution prepared, ml; and

M = mass of paper sample taken for testing, g.

B-3 DETERMINATION OF LEAD (as Pb)

B-3.1 Principle

The lead content of the sample is determined by electrothermal atomic absorption spectrometric method.

B-3.2 Apparatus

B-3.2.1 *Atomic Absorption Spectrometer* — with graphite oven technique in place of conventional burner assembly.

B-3.2.2 *Lead Hollow-Cathode Lamp or Multielement Hollow-Cathode Lamp —* For use at 283.3 nm.

B-3.2.3 *Hot Plate*

B-3.3 Reagents

B-3.3.1 *Nitric Acid* — concentrated.

B-3.3.2 *Nitric Acid* — 1:1.

B-3.3.3 *Dilute Nitric Acid* — 1:499.

B-3.3.4 *Stock Lead Solution*

Dissolve 1.599 9 g of $Pb(NO_3)_2$ in a mixture of 10 ml of concentrated HNO_3 and 100 ml of water and dilute to 1 litre. One millilitre of this solution contains 1.0 mg of lead (as Pb).

B-3.3.5 *Intermediate Lead Solution*

B-3.3.6 *Standard Lead Solution*

Dilute 100 ml of intermediate lead solution to 1 litre with dilute nitric acid (1:499). One millilitre of this solution contains 0.1 mg of lead (as Pb).

B-3.4 Procedure

B-3.4.1 *Sample Preparation*

Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

B-3.4.2 *Preparation of Test Solution*

Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for 18 ± 2 h using shaker rotating/ moving at 30 ± 2 rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size.

B-3.4.3 Transfer the extract to a 250-ml conical flask. Add 5 ml concentrated nitric acid and a few boiling chips or glass beads. Slowly evaporate on a hot plate to about 10 to 20 ml. Continue heating and adding concentrated nitric acid until digestion is complete. Wash down with water and then filter if necessary. Quantitatively transfer filtrate to a 100-ml volumetric flask, dilute to the mark and mix thoroughly.

B-3.4.4 Inject a measured portion of the digested solution into the graphite oven. Dry, char and atomize according to the preset programme. Measure the absorbance.

B-3.4.5 Prepare a reagent blank and sufficient standards containing 5.0, 7.5 and 10.0 mg/l of lead by diluting suitable volume of the standard lead solution with nitric acid (1:499) and repeat as above (**B-3.4.3**). Inject a suitable portion of each standard solution in order of increasing concentration. Analyse each standard solution and measure the absorbances.

B-3.4.6 *Calculation*

Construct a standard calibration graph by plotting the absorbance versus mg of lead concentration of each standard. Read the concentration of the sample from the graph and determine the lead content of the sample from the calibration graph using the following formula:

$$\text{Lead (as Pb), ppm} = \frac{C \times F \times 100}{M}$$

where

C = concentration of lead from the calibration curve;

F = dilution factor; and

M = mass of paper sample taken for testing, g.

B-4 DETERMINATION OF CADMIUM (as Cd)

B-4.1 Principle

The cadmium content of the sample is determined by electrothermal atomic absorption spectrometric method.

B-4.2 Apparatus

B-4.2.1 *Atomic Absorption Spectrometer* — with graphite oven technique in place of conventional burner assembly.

B-4.2.2 *Cadmium Hollow-Cathode Lamp or Multielement Hollow-Cathode Lamp* — For use at 228.8 nm.

B-4.2.3 *Hot Plate*

B-4.3 Reagents

B-4.3.1 *Nitric Acid* — Concentrated.

B-4.3.2 *Nitric Acid* — 1:1.

B-4.3.3 *Dilute Nitric Acid* — 1:499.

B-4.3.4 *Stock Cadmium Solution*

Dissolve 1.0 g of pure cadmium metal in minimum quantity of concentrated nitric acid and dilute to 1 litre with distilled water. One millilitre of this solution contains 1 mg of cadmium (as Cd).

B-4.3.5 *Intermediate Cadmium Solution*

Add 1 ml of concentrated nitric acid to 50 ml of stock solution and dilute to 1 litre with distilled water. One millilitre of this solution contains 50 µg of cadmium (as Cd).

B-4.3.6 *Standard Cadmium Solution*

To 10 ml of cadmium intermediate solution add 1 ml of concentrated nitric acid and dilute to 1 litre with distilled water. One millilitre of this solution contains 0.5 µg of cadmium (as Cd).

B-4.4 Procedure

B-4.4.1 *Sample Preparation*

Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

B-4.4.2 *Preparation of Test Solution*

Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for 18 ± 2 h using shaker rotating/ moving at 30 ± 2 rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size.

B-4.4.3 Transfer the extract to a 250-ml conical flask. Add 5 ml concentrate nitric acid and a few boiling chips or glass beads. Slowly evaporate on a hot plate to about 10 to 20 ml. Continue heating and adding concentrated nitric acid until digestion is complete. Wash down with distilled water and then filter if necessary. Quantitatively transfer filtrate to a 100-ml volumetric flask, dilute to the mark and mix thoroughly.

B-4.4.4 Inject a measured portion of the digested solution into the graphite oven. Dry, char and atomize according to the preset programme. Measure the absorbance.

B-4.4.5 Prepare a reagent blank and sufficient standards containing 5.0, 7.5 and 10.0 mg/l of cadmium by diluting suitable volume of the standard cadmium solution with nitric acid (1:499). Inject a suitable portion of each standard solution in order of increasing concentration. Analyse each standard solution and measure the absorbances.

B-4.4.6 *Calculation*

Construct a standard calibration graph by plotting the absorbance versus mg of cadmium concentration of each standard. Read the concentration of the sample from the graph and determine the cadmium content of the sample from the calibration graph using the following formula:

$$\text{Cadmium (as Cd), ppm} = \frac{C \times F \times 100}{M}$$

where

C = concentration of cadmium from the calibration curve;

F = dilution factor; and

M = mass of paper sample taken for testing, g.

B-5 DETERMINATION OF PENTACHLOROPHENOL (PCP)

B-5.1 Principle

PCP is extracted with acetone by Soxhlet extraction. Acetone extract is evaporated to dryness and subjected to acetylation. The acetylated PCP is determined quantitatively by Gas Chromatograph-Electron Capture Detector (GC-ECD).

B-5.2 Apparatus

B-5.2.1 *Round Bottom Flask* — 250 ml.

B-5.2.2 *Soxhlet Extractor*

B-5.2.3 *Water Bath*

B-5.2.4 *Separating Funnels* — 60 ml and 100 ml.

B-5.2.5 *Injection Syringes* — 1 μl, 5 μl and 10 μl.

B-5.2.6 *Gas Chromatograph with ECD* — Capillary columns.

B-5.2.7 *Glass Columns* — Length 20 cm, internal diameter 12 mm filled with 5 cm of silica gel having particle size 63 μm × 200 μm.

B-5.3 Reagents

B-5.3.1 *Acetone*

B-5.3.2 n-*Hexane*

B-5.3.3 *Acetic Anhydride*

B-5.3.4 *Sodium Sulphate Anhydrous*

B-5.3.5 *PCP Stock Solution*

Dissolve 10 mg of pentachlorophenol in 100 ml of acetone. One millilitre of this solution contains 0.1 mg of pentachlorophenol.

B-5.3.(*PCP Standard Solution*

Dilute 10 ml of stock solution with acetone to 100 ml. One millilitre of this solution contains 0.01 mg of pentachlorophenol.

B-5.3.7 *Internal Standard Stock Solution*

Dissolve 1 g of 2, 4 dibromophenol in 1 l of acetone. One millilitre of this solution contains 1 mg of dibromophenol.

B-5.3.8 *Internal Standard Solution*

Dilute 1 ml of the stock solution (**B-5.3.7**) with acetone to 100 ml. One millilitre of this solution contains 10 µg of dibromophenol.

B-5.4 Procedure

B-5.4.1 Determine the moisture content of the sample as given in 9 of IS 1060 (Part 1) : 1966.

B-5.4.2 Weigh accurately about 1 g of the paper sample pieces up to two decimal places and put into a thimble. Extract with about 150 ml of acetone by Soxhlet extraction for 6 h. Filter the acetone extract, dry over anhydrous sodium sulphate, and evaporate under vacuum to a small volume approximately to 5 ml and cool.

B-5.4.3 *Clean Up*

Transfer the extract (**B-5.4.2**) to the silica gel packed coloumn and elute with about 25 ml of *n*-hexane at the rate of 2 ml/min. Collect the eluent in a flask. Dry over anhydrous sodium sulphate and evaporte nearly to dryness.

B-5.4.4 Treat the residue (**B-5.4.3**) with 1 ml of acetic anhydride, and heat on a water bath for about 30 min. Remove the flask from the water bath and cool the acetylated product. Transfer the content in a separating funnel and add 10 ml of hexane and 5 ml distilled water. Shake well for 2 minutes and let the layers be separated. Collect the hexane layer, dry over anhydrous sodium sulphate and evaporate nearly to dryness. Cool for at least 10 minutes, add 1 ml of internal standard solution and adjust the volume to 5.0 ml with *n*-hexane.

B-5.4.5 Inject 2 µl of the solution into the Gas Chromatograph. Record the peak size in area and peak height units. If peak response exceeds linear range of the system, dilute the concentration of the extract and reanalyze.

B-5.4.6 *Calibration*

Prepare three calibration standards from the PCP standard solutions. Add 1 ml of internal standard solution and follow the steps as above (**B-5.4.3** to **B-5.4.5**).

15

Tabulate peak height or area responses against calculated equivalent mass or underivatized pentachlorophenol injected. Prepare a calibration curve.

B-5.5 Calculation

Determine the PCP content of the sample from the calibration graph using the following formula:

$$\text{PCP content, mg/kg on dry matter} = \frac{A \times B \times V_i \times 100}{M \times C \times V_t \times (100 - X)}$$

where

A = PCP content in μg from the graph;
B = total volume of hexane extract before derivetization, ml;
V_i = volume of eluent injected, ml;
M = mass of the paper sample taken for testing, g;
C = volume of hexane extract carried through derivatization, ml;
V_t = volume of total eluent, ml; and
X = moisture content, percent by mass.

B-6 DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs)

B-6.1 Principle

PCB is extracted with boiling ethanolic potassium hydroxide solution. An aliquot of the extract is mixed with distilled water and subjected to extraction with hexane. The PCB content is determined quantitatively by GC-ECD by comparing the pattern of the peaks with the pattern of a suitable technical PCB.

B-6.2 Apparatus

B-6.2.1 *Erlenmeyer Flask* — 200 ml.

B-6.2.2 *Water Bath*

B-6.2.3 *Separating Funnels* — 60 ml and 100 ml.

B-6.2.4 *Injection Syringes* — 1 μl, 5 μl and 10 μl.

B-6.2.5 *Gas Chromatograph with ECD* — Capillary columns.

B-6.2.6 *Glass Columns* — Length 20 cm, internal diameter 12 mm filled with 5 cm of silica gel having particle size 63 $\mu m \times 200 \mu m$.

B-6.3 Reagents

B-6.3.1 *Ethanolic Potassium Hydroxide Solution*

Prepare 1 N ethanolic potassium hydroxide solution by dissolving the required amount of potassium hydroxide in absolute ethanol that has been purified as follows:

Dissolve 1.5 g of silver nitrate in 3 ml of water and add it to one litre of alcohol. Dissolve 3 g of potassium hydroxide in the smallest amount of hot distilled water, cool, and add it to the silver nitrate solution. Shake thoroughly, allow the solution to stand for at least 24 h, filter and distill.

NOTE — Absolute alcohol denatured with 10 percent by volume of methanol may also be used.

B-6.3.2 n-*Hexane*

B-6.3.3 *Sodium Sulphate Anhydrous*

B-6.3.4 *PCB Stock Solution*

Dissolve 10 mg of any PCB technical in 100 ml of hexane. One millilitre of this solution contains 0.1 mg of PCB.

B-6.3.5 *PCB Standard Solution*

Dilute 10 ml of stock solution with hexane to 100 ml. One millilitre of this solution contains 0.01 mg of PCB.

B-6.3.6 *Internal Standard Stock Solution*

Dissolve 1 g of 2, 4 dibromophenol in 1 l of *n*-hexane. One millilitre of this solution contains 1 mg of dibromophenol.

B-6.3.7 *Internal Standard Solution*

Dilute 1 ml of the stock solution (**B-6.3.6**) with hexane to 100 ml. One millilitre of this solution contains 10 μg of dibromophenol.

B-6.4 Procedure

B-6.4.1 Determine the moisture content of the sample as given in **9** of IS 1060 (Part 1) : 1966.

B-6.4.2 Weigh accurately about 1 g of the paper sample pieces up to two decimal places and take into a 200-ml Erlenmeyer flask. Add 50 ml of ethanolic potassium

17

hydroxide solution and heat under reflux condenser for 4 h, filter and dilute to the mark with alcohol in a 250-ml volumetric flask. Take an aliquot portion of the test solution in a seperating funnel, add sufficient distilled water and 15 ml hexane. Shake well for 5 minutes and wait for 15 minutes. Collect the hexane phase.

B-6.4.3 Filter the hexane extract. Dry the filtrate over anhydrous sodium sulphate, and evaporate under vacuum to approximately 5 ml. Take the residue for clean up.

B-6.4.4 *Clean Up*

Transfer the hexane extract (**B-6.4.3**) to the silica gel packed column and elute with about 25 ml of *n*-hexane at the rate of 2 ml/min. Collect the eluent in a flask and evaporate to a small volume. Add 1 ml of internal standard solution and made up to 10 ml with hexane.

B-6.4.5 Inject 2 μl of the solution to the Gas chromatograph. From the peaks obtained PCB and Internal Standard are identified by their retention times as well as relative retention time.

B-6.4.6 *Calibration*

Prepare three calibration standards from the PCB standard solutions and follow the steps as above (**B-6.4.3** to **B-6.4.5**). Tabulate peak height or area responses against calculated equivalent mass of underivatized pentachlorophenol injected. Prepare a calibration curve.

B-6.5 Calculation

Determine the PCB content of the sample from the calibration graph using the following formula:

$$\text{PCB content, mg/kg on dry matter} = \frac{A \times V_i \times 100}{M \times V_t \times (100 - X)}$$

where

A = PCB content in μg from the graph;

V_i = volume of eluent injected, ml;

M = mass of the paper sample taken in g;

V_t = volume of total eluent, ml; and

X = moisture content, percent by mass.

(CHD 16)